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Original article

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Abstract:

Vibrational properties of wood are affected by several parameters, of which extractives can be one of the most important ones. Wood for European musical instruments has been often studied, but traditional Middle Eastern ones had been left unnoticed. In this study white mulberry (*Morus alba* L.), the main material for long-necked lutes in Iran, was extracted by five solvents of various polarities (water included). Free-free bar forced vibrations were used to measure longitudinal (L) loss tangent (tanδ), storage (elastic) modulus (E') and specific modulus (E'/γ) in the acoustic range. Their anisotropy between the 3 axes of orthotropy was determined by DMA (dynamic mechanical analysis). Native wood had a quite low E_L'/γ but its tanδ was smaller than expected, and the anisotropy of tanδ and E'/γ was very low. Removal of extractives caused tanδ to increase and moduli to decrease. Acetone, the most effective solvent on damping despite a moderate extraction yield, increased tanδ_L by at least 20% but did not modify E'/γ as much. When used successively, its effects masked those of solvents used afterwards. Anisotropy of E'/γ was nearly unchanged after extraction in methanol or hot water, while tanδ was much more increased in R than in T direction. Results suggest that in white mulberry, damping is governed more by nature and localization of extractives rather than by their crud abundance.

Introduction:

Wood has always been considered as a multifunctional material. Each of its versatile properties makes it appropriate for a particular final use. Wood historical use in musical instruments is in relation with its dynamic mechanical, or vibrational, properties, including mechanical damping (loss tangent) tanδ, storage (elastic) modulus E' and specific modulus E'/γ. As a quasi-orthotropic material at the macroscopic scale, wood has different properties in longitudinal (L), radial (R) and tangential (T) directions (Backman and Lindberg[1]; Nairn[2]; Brémaud et al[3]). In the case of wood used for Western classical string instruments, it is generally accepted that a low tanδ and high E'/γ in axial direction, and a high anisotropy, are convenient for obtaining a soundboard of good quality. However, few studies have addressed the vibrational properties of wood used in Middle-East instruments. String instruments in Iran can be traced back to 970 B.C. Long-necked lutes *Tar*, *Setar* and *Kamancheh* are specifically made from white mulberry (*Morus alba* L.), which is also used for the Japanese short-necked lute *Biwa* (Yoshikawa[4]).

Sources of variations in dynamic mechanical properties of wood include cell arrangement and grain angle, microfibril angle within the cell wall, proportions of constitutive polymers, as well as the presence of additional compounds (Ono and Norimoto[5,6]; Obataya et al[7]; Noda et al[8]; Bucur[9]). Although in lower quantities than cell wall constitutive polymers, extractives can have a considerable effect on mechanical and damping properties of wood. Exceptionally low damping of some musically important woods was somehow correlated with their extractives. Extractives impact can be studied either through correlations between their amounts and vibrational properties variation resulting by their removal, or by the re-injection of extracts into a "neutral" wood. Methanol extraction increased the tanδ of heartwood by 15 to 37% in red cedar (*Thuja plicata*) and up to 60% in African Padauk *(Pterocarpus soyauxii*), while E'/γ

slightly decreased (Yano[10]; Brémaud et al.[11]). Impregnation of water-soluble extractives of pernambuco (*Caesalpinia echinata*) into spruce decreased tanδ by a half (Matsunaga et al.[12]). The formation of hydrogen bonds between extractives and cell wall matrix was proposed as a possible cause of tanδ reduction (Matsunaga et al.[13]). Impregnation of Sitka spruce with isolated key chemical compounds from muirapiranga (*Brosimum rubescens*), a Moraceae as is mulberry, decreased tanδ by up to 25%, both in axial and radial directions. This was explained by motion restriction of matrix macromolecules due to extractives entering (Minato et al.[14]). Contrary to these studies, water-soluble extractives of Reed (*Arundo dunax* L.) increased both tanδ and E' (Obataya and Norimoto[15]; Obataya et al.[16]). Extractives can also change wood equilibrium moisture content (EMC) (Hernandez[17]). Higher EMC induces higher tanδ and lower E'/γ (Akitsu et al.[18]; Obataya et al.[19]). The potential effect of extractives on vibrational properties could come from their chemical structure, cellular location and relation with basic polymers (Matsunaga et al.[13]; Brémaud et al.[11]). Removing extractives using solvents of different polarities could give a first idea about their polarity and possible cellular location.

Our work aims at evaluating the dynamic mechanical properties of white mulberry and understanding the possible contribution of extractives. Basic properties of samples from different regions of Iran were compared in a previous study (Se Golpayegani[20]; Pourtahmasi and Se Golpayegani[21]). In the present article, mulberry wood was extracted in five solvents of different polarities. In order to separate the overlapping effect of various solvents, they were used both as independent extracting agents, and in a successive order on the same specimens, to determine changes in acoustical properties along the grain. Furthermore, the effects on the anisotropy of tanδ, E' and E'/γ were also studied after extraction by the two solvents with highest yields.

Material and Methods

Material

Mulberry trees were cut in Iran and kept for one month in outdoor conditions. Wood was selected there by a professional instrument maker. Several 500×50×50 mm (L×R×T) rods were cut from the middle of the heartwood and sent to France, in three batches within one year. The 1st batch (used for powder samples and independent extractions on vibrational specimens) and 2nd batch (successive extractions on vibrational specimens) came from the same tree; the 3rd (specimens for DMA) came from another tree.

A portion of each rod was ground, the fraction passing through 40 mesh and retained on 60 mesh sieves was kept for powder extractions (TAPPI standard method T207_cm[22]). For vibrational tests, the rods were cut into approx. 200 axial specimens of dimensions 150×15×2 mm^3 (L×R×T), from which 77 specimens were kept for extractions and controls (Table 1). For DMA (Dynamic Mechanical Analysis), specimens were cut to a cross section of 4×3 mm^2 and a length of 42-48 mm (Fig. 1). 14 specimens were prepared in each direction of orthotropy leading to a total number of 42.

Conditioning and physical measurements

The specimens were oven dried at 60˚C for 48 h. This procedure ensured that the equilibrium was reached in adsorption. Residual moisture content, as compared with oven drying at 103°C, was around 1.36±0.49%. Specimens were then kept for three weeks in controlled conditions of 20±2˚C and 65±5% RH. This procedure was repeated before any mechanical measurement. Specific gravity γ (both oven dried and air dried) and EMC were recorded. EMC of native wood was of 7.9±0.8%.

Although mechanical measurements could not be run in a climatic chamber, mass change was small: -0.3±0.4% for native state specimens, both during vibrational and DMA tests. For treated specimens, mass change was negligible (-0.1±0.1%) during vibrational tests, and less than during DMA tests (-0.6±0.7%).

Measurement of dynamic mechanical properties

Axial dynamic mechanical properties were measured both by vibrational tests and by DMA, while anisotropy was studied only by DMA.

Non-contact forced bending vibrations of free-free bars

A frequency scan was imposed through an electric magnet facing a tiny steel plate (15-20 mg, a negligible additional weight) glued at one end of each specimen. A laser triangulation sensor measured the displacement. A program developed with LabView® software (Brémaud[23]) monitored vibration emission and detection. Specific elastic (storage) modulus (E_L'/γ) was calculated from the first resonance frequency by the Euler-Bernoulli formula and elastic modulus (E_L') was obtained by multiplying E_L'/γ by specific gravity (γ). Damping coefficient was measured using both bandwidth at half power in the frequency domain (or quality factor) and logarithmic decrement of amplitude in the time domain after stopping the vibration. Both measurements shall be equivalent to loss tangent $\tan\delta$, if $\tan\delta << 0.1$, which is the case for air-dry wood in this temperature/frequency range. Three repetitions were made for each specimen. Resonance frequencies were in the range of 200-400 Hz.

DMA (Dynamic mechanical analyzer)

The viscoelastic behavior of specimens was measured using a BOSE® ELF3230 DMA equipped with tension/compression fatigue grips, a 22 N (±0.17% maximum error) load cell

and a high-resolution displacement sensor (1 mm range with ±0.26% maximum error). The specimens were fixed between two clamps with a working distance of 35 mm and tested in tension along their longest direction. The tests were done in purely alternative tension/compression loading controlled in displacement with an amplitude of ±0.0175 mm corresponding to a maximum tension/compression strain of ±0.02% (i.e., within the linear viscoelastic region, see Sun et al.[24]). Using BOSE WinTest® analysis software, E^* (norm of the complex modulus), E' (storage modulus), E'' (loss modulus) and tanδ (loss tangent) were calculated. Both moduli and tanδ were corrected for the stiffness of the whole apparatus (around 1785 N/mm with a negligible viscous contribution), measured using a stiff steel specimen. Frequency sweeps from 0.1 to 10 Hz were run in triplicate for each specimen, native and then treated.

Extraction procedures

Extractions used solvents of increasing polarity: hexane (HX), dichloromethane (DM), acetone (AC), methanol (ME) and hot water (HW). Most used Soxhlet extractors. Groups of specimens, with similar ranges in properties, were defined after vibrational tests in native state and subjected to different treatments (Table 1).

For extractions on powder and on solid specimens for vibrational tests, extractions were run both in an independent (or parallel) and in a successive (or serial) way. Independent extraction means that a sample is submitted to one extraction in a given solvent, and its properties are measured before and after this single treatment. In this case, hot water extraction from vibrational specimens was run (for 8h) at 70°C (not using Soxhlet). Successive extractions means that a given group of specimens is submitted to extraction, first by the less polar solvent (HX), then oven-dried (brief process: weighted, air-dry stabilized, weighted,

mechanically tested, and dried again for solid wood specimens), then extracted by the next solvent (DM), and so on. In this case, a "standard" Soxhlet extraction (<95°C) was run for water (complete description of conditions can be found in table 1).

Specimens were dried before being extracted, so that less polar solvents could not enter cell walls. Control specimens underwent only physical steps (drying and stabilization) and were measured in the same time and condition as extracted ones. For determination of extractive content, 3 g of powder were put in a cellulose cartridge, extracted for 8 h, and their oven-dry (48 h at 60°C) weight loss was measured. For solid wood, as color hardly changed during extraction, duration of 12 h was chosen. Based on the yields of extractions in vibrational specimens, ME and HW were applied as independent solvents on DMA specimens. For each anisotropic direction, 4 specimens served as controls, 5 were extracted in ME and 5 in HW (70°C, without Soxhlet). Although hemicelluloses may be partially depolymerized at moderately high temperatures for water-saturated wood (Placet et al[25]; Assor et al[26]), treatment temperatures and duration were sufficiently low to neglect such effect.

Results and Discussion

Properties of native mulberry wood

The specific gravity γ of the mulberry wood under study ranged from 0.45 to 0.61 (Fig. 2). This was significantly lighter than wood from different regions in a previous study (Se Golpayegani[20]). The 1st batch (used for independent extractions) was significantly denser than the 2nd one (used for successive extractions), although they came from the same tree. The 3rd batch (used for DMA), which came from a different tree, covered a broad range, but its

178 average γ was not significantly different from the 2nd batch. Differences in density were not
179 clearly related to those in vibrational properties (Fig. 3).

180 The specific dynamic modulus in L direction of all studied batches of wood was rather low
181 (Fig. 3) and variable (11-18 GPa). Although dispersion was quite large, $\tan\delta_L$ was negatively
182 related to E'_L/γ. However, all batches generally had a lower $\tan\delta_L$ than the "standard trend"
183 from Ono and Norimoto [5, 6], a statistical relationship obtained on 20 softwoods and 30
184 hardwoods that can be considered as a reference. In the 1st and 3rd batches $\tan\delta_L$ was in
185 average 6% and 11% lower than "standard", but with a higher dispersion than in 2nd batch and
186 in wood from a previous study (Se Golpayegani[20]), for which $\tan\delta$ was always within the
187 lower range (21% and 23% lower than the standard).

188 $\tan\delta_L$ measured with DMA method was higher than that measured with free-free vibration.
189 However, it exhibited a decreasing trend against frequency, so that the difference could be
190 attributed to the higher frequency of the free-free vibration. This decrease of $\tan\delta$ with
191 frequency was also observed in other directions, as well as a slight increase of elastic moduli.
192 However, the anisotropic ratios remained constant in the observed frequency range. In the
193 following analysis, only the values measured at 10 Hz will be used. Ordering of the different
194 $\tan\delta$ in the 3 principal directions was the same as previously reported, i.e., $\tan\delta_T > \tan\delta_R >$
195 $\tan\delta_L$ (Ono and Norimoto[27]). However, damping anisotropies between the three main
196 directions were small: R/L≈ 1.03, T/L≈ 1.34, and T/R≈ 1.30. These values were lower than
197 those collected from several studies by Brémaud et al.[3] in which the average ratios were of 2.7
198 (R/L), 2.9 (T/L) and 1.14 (T/R) for hardwoods. Similarly, the anisotropy of E' agrees with
199 well-known relationships, i.e., $E'_L >> E'_R \geq E'_T$, but actual values of ratios (L/R ≈ 4, L/T ≈ 8
200 and R/T ≈ 1.9) were in the lower range of anisotropy compared with literature reviews (e.g.,
201 Guitard and El Amri[28],Nairn[2]). It can be noted that mulberry has a low longitudinal E'/γ and a

202 very reduced anisotropy when compared with resonance spruce that is used for top plates of
203 Western string instruments, and is closer to maple (used for back and sides) in this respect.

204 ***Yield of extraction***

205 In wood powder (Fig. 4a), approximately the same cumulated amount of extractives was
206 removed when applying solvents independently or successively. In solid wood (Fig. 4b), on
207 the contrary, cumulated weight losses differed between these two extraction procedures,
208 suggesting some structural effect on the accessibility to various solvents. This was supported
209 by different extraction yields from axial, radial and tangential DMA specimens: 9, 14 and
210 18% respectively for ME.

211 Less polar solvent HX and DM, which should reach only the lumen, removed small and
212 comparable amounts (1.6% and 1.7%) when used independently on powder. Their yields were
213 much smaller in solid wood. When DM was used after HX, its yield was very low, suggesting
214 that HX had already removed most of extractives accessible to apolar solvents. Similarly, AC
215 had a smaller yield when used after HX and DM, suggesting that when used independently, it
216 also removed compounds from lumens. Although AC and ME are thought to be able to
217 solubilize similar types of compounds, ME caused the most exhaustive extraction from solid
218 wood: 6.2% when used independently. Its efficiency was even increased when used after AC
219 (7.14% from solid wood). On the contrary, in powder, HW had the highest yield (8.31%)
220 when used independently, while it had a much smaller yield on solid. This suggests that in
221 solid wood, polar extractives are more easily removed by ME than by HW, as the final
222 cumulated yields were however similar between powder and solid.

223 ***Extraction effects on E_L'/γ***

As extractions change the mass and specific gravity (Table 2, Table 3) of wood specimens, E_L'/γ values should be corrected for the contribution of extractives to γ:

$$\left(\frac{E_L'}{\gamma}\right)_c = \left(\frac{E_L'}{\gamma}\right)_{ap} \times \frac{\gamma_{ap}}{\gamma_{ef}}, \quad (1)$$

where $(E_L'/\gamma)_{ap}$ and γ_{ap} are the apparent measured values, and $(E_L'/\gamma)_c$ is the corrected value taking into account the specific gravity γ_{ef} after the most effective extraction (ME). $(E_L'/\gamma)_c$ should be more representative of an "extractives-free cell wall" modulus, and appeared 0.01 to 1.2 GPa higher than the apparent one (Table 2, Table 3).

Corrected E_L'/γ gradually decreased due to extractions. HX, DM, AC, ME and HW modified E_L'/γ by 0.2%, -0.6%, -5%, -11% and -1.8% respectively after independent extractions (Fig. 5a). Cumulated changes (i.e. relative to native state) after successive extractions followed a similar trend but with a bigger amplitude (Fig. 5b). E_L' shows a similar gradual decrease. Decrease in E_L'/γ appears nearly proportional to weight loss for different solvents (Fig. 5b), which suggests that extractives removed by AC, ME and HW bulked the cell wall matrix, and affected elasticity independently of compounds removed by these various solvents.

Changes in tanδ_L related to weight loss

Figure 6 shows relative changes in tanδ_L caused by each solvent either used individually or after others (successively). Apolar solvents (HX and DM) removed small amounts (approx. 1%) of extractives, probably from the lumens, resulting in small changes in tanδ_L. In successive extraction, however, DM increased tanδ_L by 14%, which might be linked to previous exposition to HX. On the other hand, in another species (Padauk), DM was able to

244 slightly attain cell wall and increase tanδ_L of approx. 20% for approx. 3% extracts removed
245 (Brémaud et al.[11]).

246 More polar solvents (AC, ME and HW) are expected to remove higher proportions of cell
247 wall extractives, more susceptible to alter tanδ_L. However, the highest change in tanδ_L
248 resulted from AC extraction (≈+20% independently or used after DM, ≈+38% for cumulated
249 successive effect), although its weight loss was moderate. ME and HW, despite their higher
250 yields, had smaller effects on tanδ_L both in independent and in successive-cumulated order.
251 When used after AC, ME even reduced tanδ_L lower than the value for the previous state (AC-
252 extracted, Fig. 6b). On the contrary, in other species, methanol extractives often have a high
253 "anti-damping" effect in wood (Yano[10]; Minato et al.[14]; Brémaud et al.[11]). Acetone has also
254 been used recently in this kind of study, on two tropical species (Brémaud[23]; Brémaud et
255 al.[11]). It was more efficient than ME in one species, and less in the other. The contrasted
256 effects observed in mulberry could be explained by different hypotheses: (i) AC reached all
257 extractives able to alter damping, leaving only "inactive" compounds for subsequent ME or
258 HW extractions. As drying cycles could reduce tanδ_L by ≤8%, changes in tanδ_L after ME and
259 HW in successive order could partly reflect drying history of specimens. However, a smaller
260 effect on tanδ_L of ME and HW than that of AC was also observed for independent extractions,
261 without cumulated drying cycles. This brings us to the second hypothesis ; (ii) Two kinds of
262 extractives compounds would co-exist in cell walls, some decreasing, and some increasing,
263 tanδ_L in native wood. AC would have removed the first ones, so that extraction of remaining,
264 "tanδ_L raising", compounds by ME and HW would decrease again the tanδ_L of solid wood.

265 In various woods, methanol is reported to take out non-structural carbohydrates and phenolics
266 (e.g. Rowe and Conner[29]). White mulberry has quite important content of phenolic extractives
267 (De Rosso et al.[30]). In some woods, polyphenols can form part of the fiber cell wall matrix

(Kleist and Bauch[31]), while in some other woods, they may be either preferentially in vessels, parenchyma walls (Dünisch et al.[32]), or too much condensed to enter the cell wall substance (Koch and Kleist[33]). In the later hypothesis, ME might have removed some lumen deposits, resulting in a lower ratio between changes in $\tan\delta_L$ and extraction yield. However, decreases in E'_L/γ after ME or HW suggested cell wall location. Thus, ME could have removed compounds such as sugars or simple phenols, which presence will increase $\tan\delta_L$ (Obayata et al.[15]; Sakai et al.[34]). A decrease in $\tan\delta_L$ is also observed after ME extraction of *Thuja plicata* sapwood – which should contain non-structural carbohydrates – whereas a completely opposite effect is observed in the heartwood of the same species (Yano[10]). In mulberry, extraction in HW, following ME, did not bring additional changes in $\tan\delta_L$, although it further removed ≈4% extractives. In *Caesalpinia echinata*, water soluble extractives decrease $\tan\delta_L$ in wood, which was first ascribed to their ability to form hydrogen bonds with cell walls components (Matsunaga et al.[12]). But other extractives, without hydroxyl groups and/or insoluble in water, also reduce $\tan\delta_L$ (Minato et al.[14]; Brémaud et al.[11]). Finally, differences in $\tan\delta_L$ might be related to changes in wood moisture content (Dunlop[35]; Obayata et al.[19]; Inokuchi et al.[36]). However, changes in EMC due to independent extractions were small (Table 2). In successive extractions (Table 3), with higher cumulated yields, EMC was more significantly increased after ME and HW. This, however, should lead to an increase in $\tan\delta_L$, instead of the observed decrease for successive use of ME and HW.

Therefore, the present results suggest the co-existence of some "tanδ – lowering" and of some "tanδ – raising" compounds in the heartwood of white mulberry. The first ones are extractable by ME but also with AC and HW, consequently independent extraction with those solvents had always resulted in an increase in $\tan\delta_L$. The latter compounds, being probably hydrophilic, are not extractable by HX, DM or AC. Thus a successive extraction using ME

and HW had reduced tanδ_L, as the "tanδ – lowering" had been already removed by previous solvents and there were only the "tanδ – raising" components left. The methodology using both independent and successive series of extraction might reveal similar trends in other species.

Changes in anisotropic properties due to extractions

Figure 7 shows the variations in E'/γ and tanδ in the three principal directions of orthotropy after extractions with HW and ME or 2 drying cycles (controls). Variations in E'/γ after ME extraction were similar in all three directions: -18% to -20%, although weight losses were different (9%, 14% and 18% for L, R and T specimens respectively). Hot water caused both lower weight losses (6-8% in L, R and T), and smaller decrease in E'/γ along L and R directions, while it was similar to ME in T direction.

Concerning tanδ, uncertainty was high in L direction, as clamping of specimens may crush wood in the softer transverse direction. Even controls showed important variations, which cannot be ascribed to physico-chemical changes. However, after ME extraction, change in radial tanδ was at least twice more important than in L (DMA estimations and changes in vibrational tests) and T direction (Fig. 7).

The quasi-isotropic effect of extractions on E'/γ is quite surprising. In previous works, extractives stiffened the transverse moduli (Yano et al.[37]; Minato et al.[14]), but not the axial one, in which the influence of the microfibrillar reinforcement predominates (Ono and Norimoto [5, 6]; Obataya et al.[7]). However, mulberry wood has a low longitudinal E'/γ, suggesting high microfibril angle, and is characterized by a very low anisotropy in native state. Effects of extractions on tanδ were clearly different in radial and tangential directions. This is interesting, as there are relatively few works on the tanδ_R/tanδ_T anisotropy and sources

315 of variations. Backman and Lindberg[1] stated that, for softwood, in tensile test latewood
316 contributes mostly to the response in tangential direction while most of the strain occurs in
317 early wood in radial direction. In mulberry, a ring-porous hardwood, the response in radial
318 tension tests would more involve vessels and rays, whose cell walls are thinner, but are also
319 susceptible to contain higher proportions of extractives (Koch[38]; Kleist and Bauch[31]; Dünisch
320 et al.[32]). Thus, the very high change in $\tan\delta_R$ after ME extraction (≈ 50%) could express a
321 bigger modification of rays and vessels, than of fibers.

322 # Conclusion

323 Dynamic mechanical properties E'/γ, $\tan\delta$, and their anisotropic ratios in the 3 principal axes
324 were measured on white mulberry wood (*Morus alba* L.), a representative raw material for
325 musical instruments in Iran. They were compared before and after extractions by different
326 solvents in order to investigate extractives effects. Results could be synthesized as follows:

327 - Mulberry wood has a quite low E'_L/γ but its damping factor ($\tan\delta_L$) is lower than
328 expected. The anisotropy of these two properties is also much lower than average.

329 - Combining two methods of extractions -using each solvent as an individual agent and
330 using solvents in a successive order on a single sampling- could suggest the
331 importance of extractives nature compared to their amount.

332 - The highest changes in $\tan\delta_L$ were not due to the most polar solvents, with highest
333 extraction yields, but to acetone. Nevertheless, acetone did not alter E'_L/γ with the
334 same intensity, suggesting that the extracted amounts or compounds were not essential
335 for wood stiffness.

- The different effects of methanol extraction on $\tan\delta_L$, between individual extraction and when used successively after acetone, suggest the existence of two types of $\tan\delta$ altering compounds in white mulberry. Some would be able to increase, some other would be able to reduce the damping, and both types would be accessible by acetone and methanol independently.

- Changes in E'/γ were nearly isotropic between the 3 principal axes of orthotropy, may be due to the very low anisotropy of mulberry in native state. On the contrary, $\tan\delta$ was much more modified in radial than in tangential direction, suggesting different responses to extractions of rays and vessels walls as compared to fibers ones.

It should be noted that even though extractives are found to definitely affect vibrational properties of *Morus alba* L., it would be enlightening to identify the most important compounds present in those extractives and how they cause changes in vibrational properties.

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Tables

Table 1: Specimens number and treatments for both independent and successive methods of extraction

Number of specimens	Solvent	Drying and stabilization condition	Number of cycles of drying and stabilization
12	Hexane (HX)	60°C for 48 h followed by 20±2˚C & 65±5% RH for 3weeks	2
12	Dichloromethane (DM)		2
12	Acetone (AC)		2
12	Methanol (ME)		2
5	Hot Water (HW, 70°C)		2
12	HX/DM/AC/ME/HW (100°C)		6
12	Controls		6
15 DMA(5L, 5R, 5T)	ME		2
15 DMA(5L, 5R, 5T)	HW (70°C)		2
12 DMA(4L, 4R, 4T)	Controls		2

Table 2: Basic statistics for differences in properties between groups submitted to independent extractions and to one cycle of drying/re-stabilization (=control).

Treatment	EMC (%)[d]		Specific gravity γ		E'_L/γ Corrected (GPa) E'_L/γ Apparent (GPa)		$\tan\delta_L$ (10^{-3})	
	Corrected Apparent							
	Native	Treated	Native	Treated	Native	Treated	Native	Treated[f]
Control[e]	9.2±0.5 8.2±0.4 (a)	9.2±0.2 *ns* 8.2±0.2 (ab)	0.556±0.027 (a)	0.553±0.030 *ns* (ab)	15.0±2.2 14.0±1.6 (b)	15.2±2.4 *ns* 14.2±1.6 (a)	8.6±0.3 (a)	7.9±0.5 ** (a)
Extracted								
HX	9.1±0.2 8.1±0.2 (a)	8.9±0.4 *ns* 7.9±0.4 (a)	0.570±0.031 (a)	0.566±0.030 ** (b)	13.5±2.0 12.3±1.4 (a)	13.7±2.0 ** 12.5±1.4 (a)	10.2±0.6 (b)	9.6±0.6 ** (bc)
DM	9.0±0.3 8.0±0.3 (a)	9.6±0.4 ** 8.6±0.3 (c)	0.560±0.031 (a)	0.557±0.031 ** (b)	14.3±2.2 13.2±1.6 (ab)	14.3±2.2 *ns* 13.3±1.6 (a)	9.6±1.0 (b)	9.3±0.8 * (bc)
AC	9.1±0.4 8.1±0.3 (a)	9.4±0.4 * 8.6±0.3 (c)	0.567±0.026 (a)	0.550±0.025 ** (ab)	14.6±1.6 13.3±1.1 (ab)	13.9±1.6 ** 13.2±1.1 (a)	8.7±0.4 (a)	9.8±0.6 ** (c)
ME	9.2±0.4 8.2±0.4 (a)	8.7±0.2 ** 8.3±0.3 (bc)	0.554±0.026 (a)	0.520±0.025 ** (a)	14.4±2.2 13.5±1.4 (ab)	12.9±2.0** 12.9±1.4 (a)	8.9±0.5 (a)	9.5±0.5 ** (bc)
HW	9.2±0.3 8.2±0.3 (a)	9.0±0.2 *ns* 8.3±0.1 (bc)	0.550±0.015 (a)	0.530±0.017 ** (ab)	14.1±1.7 13.3±1.3 (ab)	14.0±1.9 *ns* 13.7±1.7 (a)	8.5±0.5 (a)	8.6±0.6 *ns* (ab)

"Apparent" stands for measured values; "Corrected" take into account the contribution of extractives to weight (for EMC) and specific gravity (for E'/γ).

(a, b, c): homogenous subsets in one-way ANOVA at a level α:0.05 (=comparison between groups in a given column, based on measured values).

(*ns*, *, **): differences between untreated and treated properties of a given group (based on corrected values whenever applicable) in *t*-test for paired samples; **: significant at α: 0.01, *: α: 0.05, *ns*: not significant.

d) EMC relative to 103°C oven-drying. "Apparent" are measured values; "Corrected" is calculated by taking into account the total extractive content (cumulated % on powder) from 1st batch of wood (see Hernandez 2007).

e) "Treated" state for controls stands for values measured after one cycle of oven-drying followed by air-dry re-stabilization.

f) $\tan\delta_L$ are raw values, whereas $\tan\delta_L$ variations in Fig. 6 are corrected for controls.

Table 3: Basic statistics for the successive ranges in properties for the group of specimens submitted to successive extractions.

Treatment	EMC (%)[a] Corrected Apparent	Specific gravity γ	E'_L/γ Corrected (GPa) E'_L/γ Apparent (GPa)	$\tan\delta_L$ (10^{-3})
Untreated	7.0± 0.3 6.1± 0.3	0.515± 0.011	13.72± 1.47 12.99± 1.25	8.2± 0.5
HX	7.1± 0.3 *ns* 6.2± 0.2	0.512± 0.016 *ns*	13.46± 1.68 *ns* 12.82± 1.35	7.9± 0.4 **
DM	9.2± 0.2 ** 8.0± 0.2	0.515± 0.012 *ns*	13.51± 1.26 *ns* 12.78± 1.07	9.3± 0.6 **
AC	9.0± 0.4 *ns* 8.0± 0.3	0.515± 0.016 *ns*	13.53± 1.64 *ns* 12.79± 1.30	11.3± 0.8 **
ME	8.6± 0.1 ** 8.2± 0.1	0.505± 0.014 **	12.69± 1.38 ** 12.24±1.13	10.6± 0.6 **
HW	8.8± 0.1 ** 8.8± 0.1	0.488± 0.011 **	11.85± 1.23 ** 11.84± 1.05	10.2± 0.5 *

"Apparent" stand for measured values; "Corrected" take into account the contribution of extractives to weight (for EMC) and specific gravity (for E'/γ).

a) EMC relative to 103°C oven-drying. "Apparent" are measured values; "Corrected" is calculated by taking into account the total extractive content (cumulated % on solid specimens) from 2nd batch of wood (see Hernandez 2007).

(*ns*, *, **): differences between each successive extracted state in relation to the previous phase of ttreatment (based on corrected values whenever applicable) in *t*-test for paired samples; **: significant at α: 0.01, *: α: 0.05, *ns*: not significant.

Figure legends

Fig. 1. Cutting plan for vibrational and DMA specimens. Variations in length of DMA specimens were due to limitations in raw material.

Fig. 2. Box plot of specimens specific gravity (γ) for the three batches of wood under study, and for previous data on wood from different origins (Se Golpayegani[20]). (a, b, c) significantly different groups (one-way ANOVA).

Fig. 3. Relationship between E'_L/γ and $\tan\delta_L$ for 4 batches of native mulberry wood (N specimens: see Fig. 2).

Fig. 4: Comparison of weight loss due to independent and successive extraction in (a) powder, and (b) solid (vibrational) specimens. "Successive" weight losses are relative to the previous step; "independent" and "successive-cumulated" are relative to native oven-dry weight.

Fig. 5. Relationship between weight loss (%) and relative change of E'_L/γ (%) after (a): independent extractions and (b): successive extractions where changes are relative to initial state of native specimens (i.e. cumulated changes). E'_L/γ was corrected for contribution of extractives to γ.

Fig. 6. Relative changes (%) in $\tan\delta_L$ plotted against weight loss (%) for (a) independent and (b) successive extractions. Changes are relative to initial state of native specimens (independent and successive-cumulated). Arrows: order of successive extractions. Values of $\tan\delta_L$ in independent extractions are corrected for controls.

531 Fig. 7. Variations in mechanical properties after extractions measured by DMA at 10 Hz. Values for controls
532 were not deduced from treated ones.

533

Figures

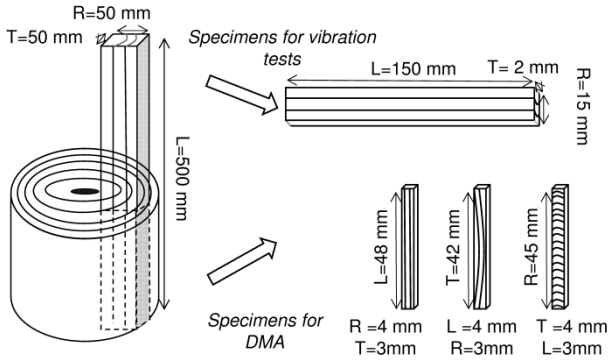
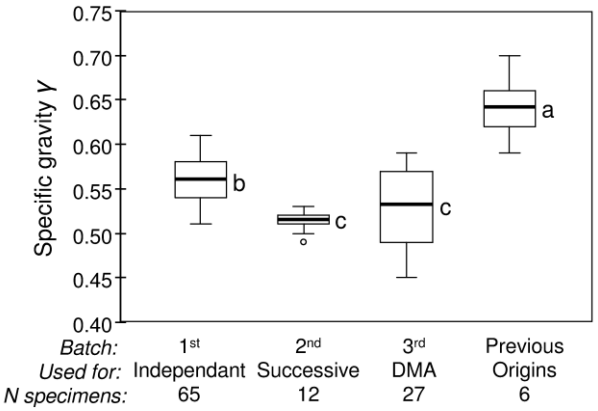


Fig. 1.



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539 Fig. 2.

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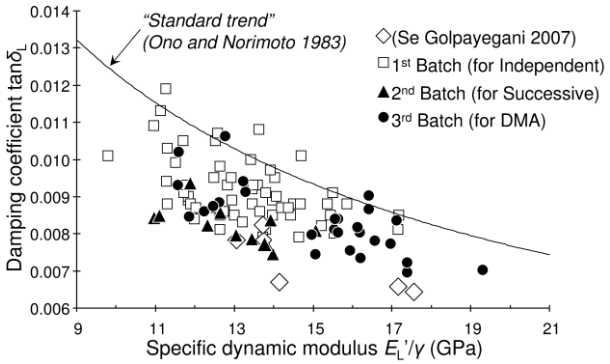
"Standard trend"
(Ono and Norimoto 1983)
◇(Se Golpayegani 2007)
□1st Batch (for Independent)
▲2nd Batch (for Successive)
●3rd Batch (for DMA)
Damping coefficient tanδL
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
9
11
13
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21
Specific dynamic modulus EL'/γ (GPa)

Fig. 3.

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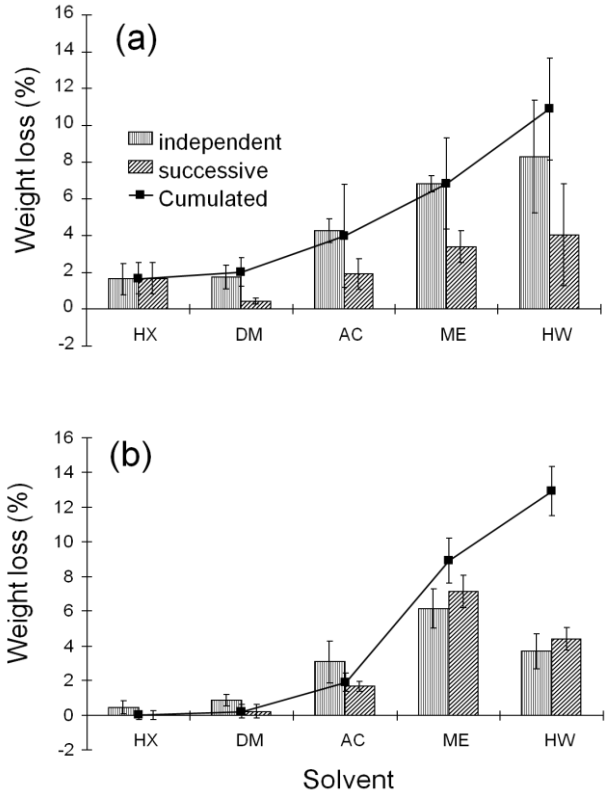

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548 Fig. 4.

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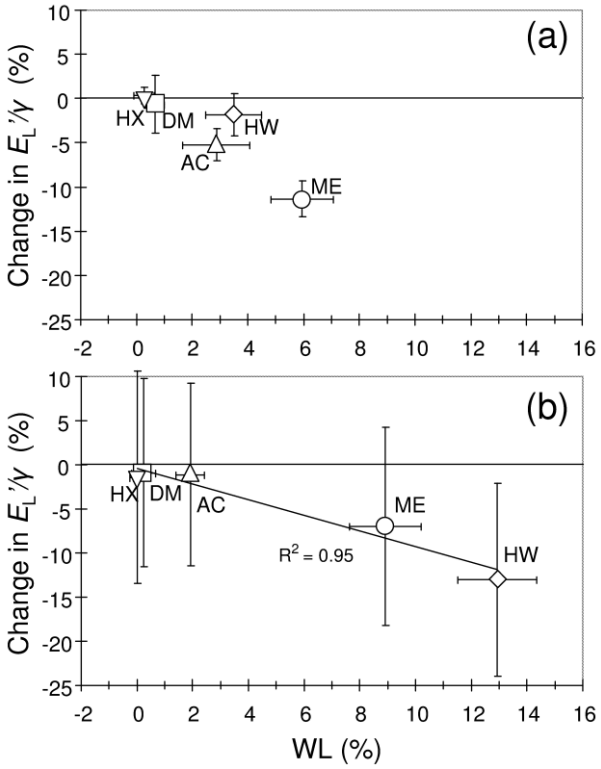

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551 Fig. 5.

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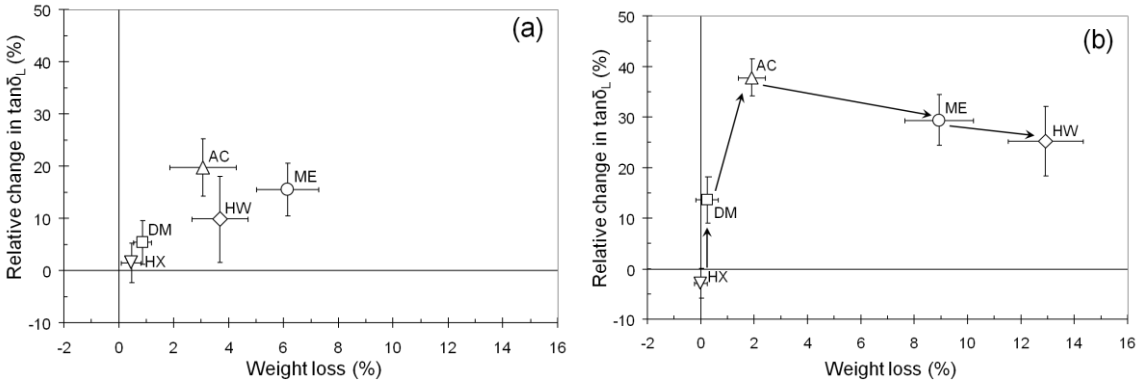

Fig. 6.

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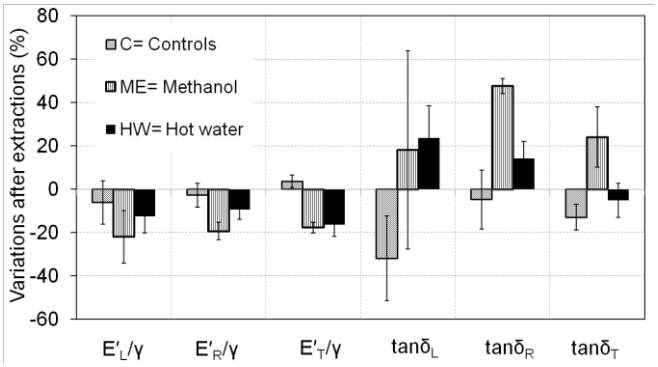

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558 Fig. 7.